Project report

Contract number: AOARD-064023

Title: Ion Beam Induced Softening of a Nanoelectromechanical Actuator

ABSTRACT

We report a novel method of engineering the mechanical properties of individual nanostructures. The method of ion irradiation serves as a non-destructive tool to manipulate the spring constant values of isolated nanorods. The slanted Si nanorods were grown by glancing angle deposition (GLAD) technique on a patterned Si(100) substrate with tungsten posts arranged in a 1μm×1μm square pattern. Another Cr nanorods sample were deposited over the Silica balls on Si substrate. The resulting slanted Si nanorods are well separated allowing us to study the mechanical properties of individual nanorods. An atomic force microscope was used in *force-distance spectroscopy* mode to determine the spring constant value of a single nanorod. The Young's modulus of the Si nanorods undergone remarkable change by 62% after the ion beam irradiation. The sample (at 80K temperature) was irradiated by 100 MeV Ag^{+8} ions at a fluence of 10^{14} ions/cm^2. The micro-Raman studies over Si nanorods before and after the irradiation show the presence of nanocrystalline regions within the Si nanorods which got amorphized after the irradiation. The ion beam induced enhancement in the amorphization and defects such as vacancies results in the softening of these nanorods. We have performed nanoindentaion studies on the Cr metal nanorods after irradiating with fluence varying from 10^{12} to 10^{14} ions/cm^2. The results show a 7 fold enhancement in the hardness value of the Cr nanorods after irradiating with fluence value of 10^{14} ions/cm^2. The results are very encouraging to use ion beam as a modification tool for tailoring the mechanical properties.

I. OBJECTIVE

Nanoelectromechanical actuators are the basic building blocks of nanoelectromechanical-systems and devices. The realization of these devices requires complete characterization of the properties and the freedom to tune them to desirable ones. This project focuses on the mechanical characterization of slanted silicon nanorods by determining the spring-constant distribution for the system and the possibility of shifting it. The spring-constant and the Young's modulus for any structure are related by a geometrical constant. Post fabrication there is not much freedom to modify the dimensions of the grown structure. However, by tailoring the Young's modulus there is a possibility to modify the spring-constants of the grown structures. The swift heavy ion (SHI) irradiation can be employed to create defect states within the nanostructures that can modify the Young's modulus and hence the spring-constant distribution of the nanorod system. Thus, the SHI

Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE
09 AUG 2007

2. REPORT TYPE
FInal

3. DATES COVERED
28-04-2006 to 09-08-2007

4. TITLE AND SUBTITLE
Ion Beam Induced Softening of a Nanoelectromechanical Actuator

5a. CONTRACT NUMBER
FA520906P0158

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
Jitendra Singh

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Indian Institute of Technology, Delhi,Physics Department,Hauz Khas, New Delhi 110016,India,IN,110016

8. PERFORMING ORGANIZATION REPORT NUMBER
N/A

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AOARD, UNIT 45002, APO, AP, 96337-5002

10. SPONSOR/MONITOR’S ACRONYM(S)
AOARD

11. SPONSOR/MONITOR’S REPORT NUMBER(S)
AOARD-064023

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited

13. SUPPLEMENTARY NOTES

14. ABSTRACT

A novel method of engineering the mechanical properties of individual nanostructures is reported. The method of ion irradiation serves as a non-destructive tool to manipulate the spring constant values of isolated nanorods. The slanted Si nanorods were grown by glancing angle deposition (GLAD) technique on a patterned Si(100) substrate with tungsten posts arranged in a 1μm?1μm square pattern. Another Cr nanorods sample were deposited over the Silica balls on Si substrate. The resulting slanted Si nanorods are well separated allowing us to study the mechanical properties of individual nanorods. An atomic force microscope was used in force-distance spectroscopy mode to determine the spring constant value of a single nanorod. The Young?s modulus of the Si nanorods undergone remarkable change by 62% after the ion beam irradiation. The sample (at 80K temperature) was irradiated by 100 MeV Ag+8 ions at a fluence of 1014 ions/cm2. The micro-Raman studies over Si nanorods before and after the irradiation show the presence of nanocrystalline regions within the Si nanorods which got amorphized after the irradiation. The ion beam induced enhancement in the amorphization and defects such as vacancies results in the softening of these nanorods. Nanoindentaion studies on the Cr metal nanorods after irradiating with fluence varying from 1012 to 1014 ions/cm2 was performed. The results show a 7 fold enhancement in the hardness value of the Cr nanorods after irradiating with fluence value of 1014 ions/cm2. The results are very encouraging to use ion beam as a modification tool for tailoring the mechanical properties.

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT **Same as Report (SAR)**	18. NUMBER OF PAGES **8**	19a. NAME OF RESPONSIBLE PERSON
a. REPORT **unclassified**	b. ABSTRACT **unclassified**	c. THIS PAGE **unclassified**			

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std Z39-18

irradiation parameters hold the key to tailor the mechanical properties of individual nanorods.

II. RESEARCH WORK CONDUCTED

a. Growth of nanostructures

The slanted silicon nanorods were grown by glancing angle deposition technique [1-3]. The vapor flux was incident at a grazing angle of 85˚ from the substrate normal to ensure significant shadowing onto a patterned Si(100) substrate. The patterns consist of tungsten posts arranged in a square lattice of 1μm spacing. The rods are 1.5 μm in length and about 220 nm in diameter. The rods are having rise angle of about 35° as shown in Figure 1.

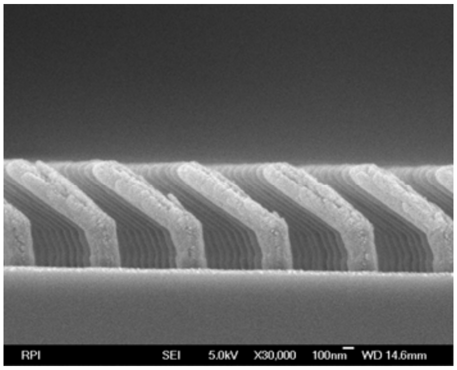

Figure 1. SEM image showing the side-view of the slanted silicon nanorods on a square-pattern

In a separate experiment Cr columnar films were grown by glancing angle dc magnetron sputter deposition technique on Si(100) patterned substrates. The patterns consist of 500 nm diameter polystyrene spheres that self-assemble in a hexagonal network on the Si substrate. These slanted chromium rod-like structures have an average length of 2 micron and diameter of 250 nm as shown in figure 2.

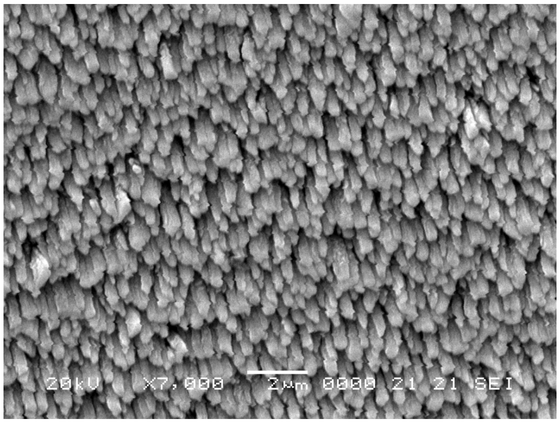

Figure 2. SEM top view image of the Cr columnar film

b. Ion beam irradiation

These slanted Si nanorods and Cr columnar structures were irradiated by 100 MeV Ag^{+8} ions at a fluence of 10^{14} ions/cm^2 by a 15 MV pelletron accelerator at the Inter-University Accelerator Centre, New Delhi. One half of the sample was masked during irradiation to preserve some pristine nanorods. The sample was maintained at liquid nitrogen temperature of about 80 K to locally freeze the defects created during ion irradiation and minimizing their diffusion in the process. The electronic (S_e) and nuclear (S_n) energy losses for the Ag ions have been calculated using SRIM as 1.05×10^4 keV/µm and 5.9×10^1 keV/µm respectively. The ratio of S_e/ S_n is about 180.

c. Force-distance (F-d) spectroscopy

After irradiation force-distance (F-d) measurements using an atomic force microscope model Veeco multimode are performed on the pristine and the irradiated nanorods. Here, force refers to the applied force and the distance is the amount by which the nanorods get deflected. The schematic of F-d measurements on an individual nanorod is shown in Figure 3. The slope of the force-distance curves yields the spring-constant values for the nanorods. The AFM cantilevers are calibrated for their sensitivity and their spring constant values prior to F-d measurements.

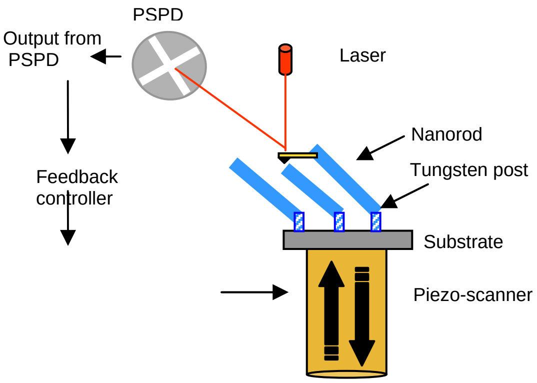

Figure 3. A schematic diagram showing the F-d measurements on a selected nanorod.

The AFM cantilevers were first calibrated for sensitivity and their spring constants. Since the cantilever deflection is measured indirectly in terms of the scanner motion (z), the exact relation between z and the cantilever deflection (y) must be known. This parameter known as sensitivity is accurately determined. The force applied by the cantilever on a nanorod is $k_{cl}\cdot y$ where k_{cl} is the spring constant of the cantilever and y is the corresponding cantilever deflection. The accuracy in applied force F thus depends upon the accuracy with which k_{cl} is known. Following the method proposed by Torii *et al.* [4], the *k*-values of the cantilevers were first calibrated. A *c* cut sapphire assumed to be infinitely hard was mounted on the AFM sample stage. For a given *z* the deflection of the cantilever (*y*) was recorded by the photo-sensitive photodetector (PSPD). For sapphire no indentation is expected. So, the entire *z* motion is transferred to the cantilever. The slope of deflection-displacement plot (*y* versus *z*) determines the sensitivity value. Whenever a fresh cantilever was used, its sensitivity was first determined. Once the sensitivity is determined, the slope of *y* versus *z* plot yields δ_{tot}. Next, a reference cantilever was mounted instead of sapphire. The slope of *y* versus *z* in this case yields δ_{CL}. The spring constant of the cantilever is then expressed as

$$k_{test} = k_{ref}\left(\frac{\delta_{tot} - \delta_{test}}{\delta_{test}}\right).$$

The cantilever is thus calibrated completely. The above procedure is followed for acquiring *y* versus *z* plot for nanorods. In the force-mode a voltage is ramped across the length of the scanner tube while the scanning halted. The scanner commences its extension-retraction cycle at a predetermined frequency which controls the loading-unloading rate of the nanorods. In one such cycle, after the contact between the tip and the nanorod is made, the cantilever attempts to deflect the rod. The cantilever deflection (*y*) depends upon the hardness of the nanorod. Unlike the case of a conventional thin film grown on a substrate where loading results in the cantilever deflection alone; neglecting any indentation effects; in the case of nanorods, the rod itself undergoes a deflection by some amount, say *d*. In case of nanorods of varying hardness, it is easier to bend a comparatively softer nanorod. Thus, the nanorod undergoes a deflection and subsequently the corresponding cantilever deflection is smaller. The opposite holds good for the harder rods. When the cantilever starts loading the selected nanorod, restoring forces appear within the nanorod due to material's elasticity. In a configuration like the one involving a nanorod and a cantilever, the PSPD reads the net deflection of the cantilever-nanorod system i.e. the sum of the deflection of the nanorod (*d*) and the deflection of the cantilever (*y*). In this case, $d = z - y$. Thus, the slope of force-distance (F-d) curve of a nanorod would yield its spring constant value. A typical force versus distance curve taken over an individual slanted Si nanorod is shown in Figure 4. The cantilevers used here were calibrated and the *k*-values were determined to be ~40 N/m. The F-d measurements were repeated on the rods and were found to be reproducible.

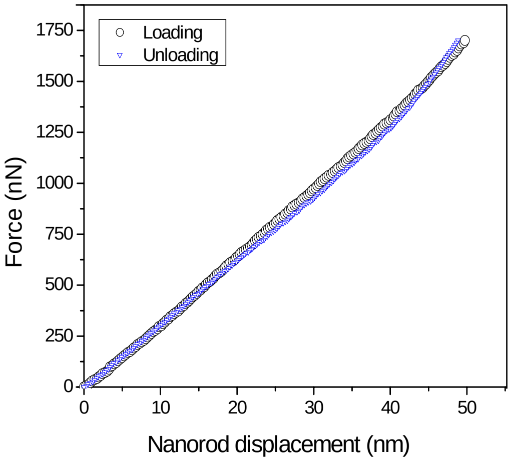

Figure 4. A typical Force-distance curve taken on an individual slanted Si nanorod obtained by AFM.

III. RESULTS AND DISCUSSION

The spring-constant value determined for pristine nanorods is about 174 N/m and for irradiated nanorods is 65 N/m showing a decrease by a factor of ~2.6. This softening of the nanorods by 62% shows that the spring-constant of the nanorods can be changed significantly. Figure 5 shows the distribution in the *k*-values for unloading case after irradiation. The *k*-values reported here have been obtained by developing a statistics of the force-distance (F-d) measurements over thirty nanorods.

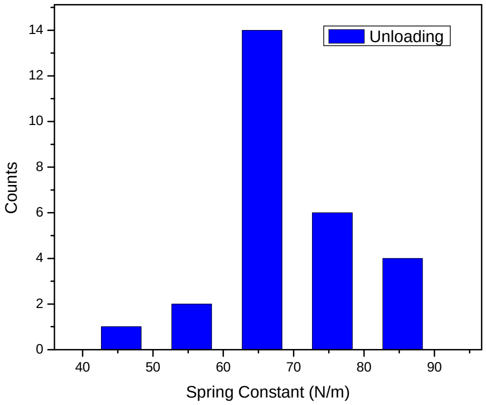

Figure 5. The distribution of *k*-values showing the unloading curve. A silicon cantilever with spring constant value of 40 N/m was used for acquiring the F-d data.

The irradiation by swift ions creates point defects within the silicon matrix. The Stopping Range of Ions in Matter software was used to estimate the range of Ag ions in Si matrix. The range of 15 μm is much more than the length of the nanorods of 1.5 μm. Hence, there is no implantation of the silver ions in the nanorods. The micro-Raman spectra collected over pristine and irradiated nanorods in Figure 6 shows that the nanorods undergo a phase change from nanocrystalline to amorphous phase. Argon ion laser at 514 nm wavelength at 50 mW power was used. An integration time of 50 s has been used. This is established by the peak shift of 509 cm^{-1} in the pristine rods to 465 cm^{-1} in the irradiated nanorods.

We propose that this softening of the nanorods is due to the defect confinement within the nanorods due to the geometrical shape of the nanorods as shown in Figure 7. The mobility of the defects created within a nanorod is different for the radial and axial directions. In the radial direction, the point defects are closely confined due to the potential felt from the boundary of the nanorod where as along the axial direction the defects have more freedom to move before coming to the rod edges. This confinement guides the point defects to align them along the nanorod length leading to the softening of the nanorods. The irradiation parameters can be explored to create defects within the material and to tune the nanorods to desirable spring-constant values.

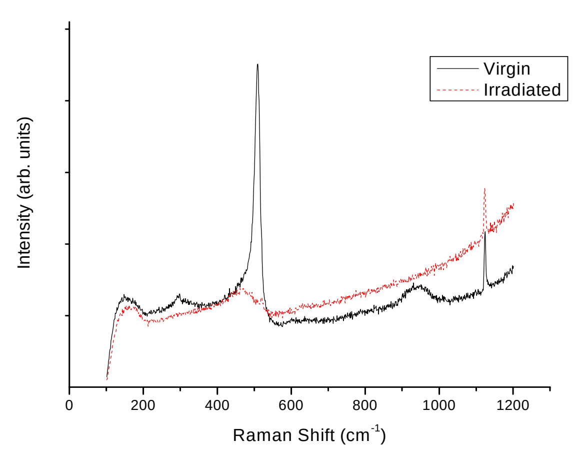

Figure 6. Micro-Raman spectra for pristine and irradiated nanorods. Spectra were required by Ar ion laser (514.4nm) at 50mW power for 50s. The peaks are labeled in Table 1.

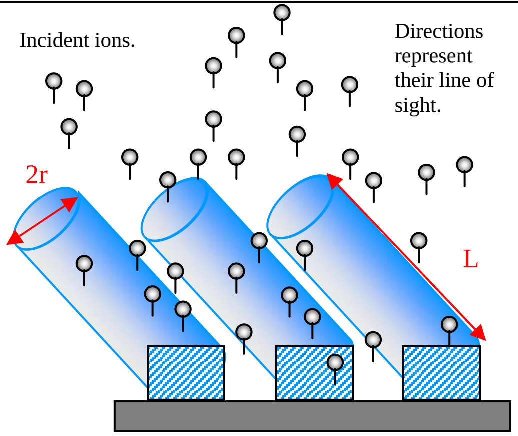

Figure 7a. Ions incident on the slanted nanorods. The spheres with a line show the incident ions with the direction of their line of sight.

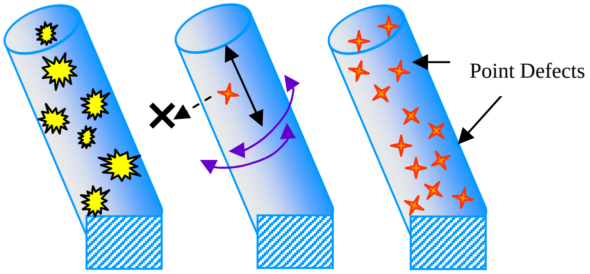

Figure 7b. Schematic of the proposed model for softening of nanorods. The yellow regions in the first rod show the nanocrystalline phase within the virgin silicon nanorods which disappear when rods get amorphized after irradiation in the third rod. The mobility of point defects formed during irradiation is different in the radial and axial directions due to the confining potential. Point defects moving towards the rod boundary in the radial direction will experience this potential more as compared to ones moving along the axial direction. This may lead to stacking of these point defects along the rod length.

We are analyzing the results for Cr columnar films. The nanohardness measurements on pristine and irradiated Cr rods have been carried out using a Berkovich diamond tip using an atomic force microscope. The representative nanoindentation curves are shown in Figure 8. The average hardness of Cr pristine rods was found to be 0.59 GPa. We have observed that the fluence of 10^{13} ions/cm^2 is not sufficient to change the hardness of Cr rods from their pristine counterparts. For higher fluences values nanohardness of Cr rods increases and becomes as much as 3.95 GPa for the fluence of 10^{14} ions/cm^2, showing about a 7 fold increase from the pristine value.

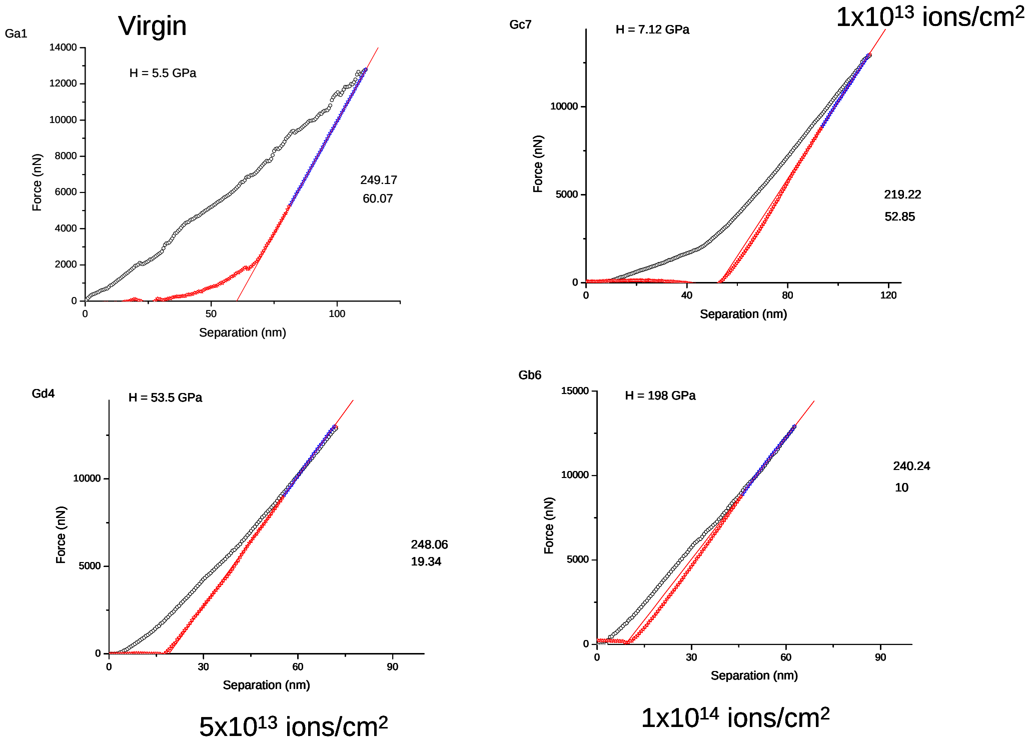

Figure 8. Nanoindentation curves for a) virgin and b-d) 100 MeV Ag^{+8} ions irradiated Cr columnar films.

The results are shown in the table. Ion beam 100 MeV Ag^{+8}

Ion fluence (ions/cm^2)	Hardness (GPa)
Virgin	0.59
1x10^{13}	0.72
5x10^{13}	2.33
10^{14}	3.95

IV. REFERENCES

1. “Physical properties of nanostructures grown by oblique angle deposition” J.P. Singh, T. Karabacak, D.-X. Ye, D.-L. Liu, C. Picu, T.-M. Lu, and G.-C. Wang, J. Vac. Sci. Technol. B, 23, 2114 (2005).
2. “Metal coated Si springs: nanoelectromechanical actuators” J.P. Singh , D.-L. Liu, D.-X. Ye, R.C. Picu, T.-M. Lu and G.-C. Wang, Appl. Phys. Lett. 84, 3657 (2004).
3. “Scaling During Shadowing Growth of Isolated Nano-Columns” T. Karabacak, J.P. Singh , Y. –P. Zhao, G. –C. Wang, and T.M. Lu, Phys. Rev. B 68, 125408 (2003).
4. A. Torii, M. Sasaki, K. Hane, S. Okuma, Meas. Sci. Technol. 7, 179 (1996).

V. PUBLICATIONS

1. “Swift heavy ion induced softening of slanted Si nanorods”, Rupali Nagar, T.-M. Lu, F. Singh, B.R. Mehta and J.P. Singh, submitted to Journal *Small*
2. “Effect of swift heavy ion irradiation on the hardness of chromium columnar films”, Rupali Nagar, S. Kesaprgada, D. Gall, V. Ganesan, B. R. Mehta and J. P. Singh, American Vacuum Society AVS 54th International conference, Oct. 14-19, 2007, Seattle, WA.
3. “Ion beam induced softening of Si nanorod structures” Rupali Nagar and J.P. Singh, presented at International conference on experimental condensed matter physics, *Advanced Nanomaterials conference* (ANM 2007), IIT Mumbai, India, Jan. 8 -10, 2007.